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Castellaroite, $\text{Mn}_2+3(\text{AsO}_4)_2 \cdot 4.5\text{H}_2\text{O}$, a new mineral from Italy related to metaswitzerite.

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(Article begins on next page)

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Abstract:	<p>Castellaroite (IMA2015-071), $\text{Mn}_{2+3}(\text{AsO}_4)_2 \cdot 4.5\text{H}_2\text{O}$, is a new secondary arsenate mineral from the Monte Nero mine, Rocchetta Vara, La Spezia, Liguria, Italy and the Valletta mine, near Canosio, Cuneo, Piedmont, Italy. It crystallized from As- and Mn-rich hydrothermal fluids in an oxidizing environment. At Monte Nero, it is associated with coralloite, manganohörsite, rhodochrosite, sarkinite, sterlinghillite, strashimirite and wallkilldellite. At Valletta, it is associated with braccoite, hematite, manganberzeliite, orthoclase and tiragalloite. Castellaroite occurs as thin blades, flattened on [001], striated and elongated parallel to [100] and exhibiting the forms {110}, {012} and {001}. The mineral is colourless and transparent with a vitreous to silky lustre and white streak. Crystals are flexible with a curved fracture, and one perfect cleavage on {001}. The Mohs' hardness is 2½. The measured and calculated densities are 3.14(2) g·cm⁻³ and 3.164 g·cm⁻³, respectively. The mineral is easily soluble in dilute HCl at room temperature. Optically, castellaroite crystals are biaxial (-), with $\alpha = 1.644(1)$, $\beta = 1.662(1)$ and $\gamma = 1.667(1)$ (white light); $2V = 57(1)^\circ$; dispersion $r < v$, moderate; the optical orientation $Y = b$; $Z \approx a$. The Raman spectrum is dominated by features corresponding to the AsO_4 group and also confirms the presence of H_2O. Electron microprobe analyses gave the empirical formula $\text{Mn}_{2+3.02}(\text{As}_{1.94}\text{P}_{0.06})\Sigma 2.00\text{O}_{12.5}\text{H}_{8.96}$, based on 12.5 O apfu. Castellaroite is monoclinic, $P2_1/n$, with the unit cell parameters: $a = 8.7565(8)$, $b = 13.4683(13)$, $c = 18.652(2)$ Å, $\beta = 94.876(7)^\circ$, $V = 2191.7(4)$ Å³ and $Z = 8$. The eight strongest lines in the X-ray powder diffraction pattern are [dobs/Å (I) (hkl)]: 10.90(100)(011), 9.27(67)(002), 6.97(42)(-111), 3.323(47)(multiple), 3.043(87)(-134,204,-232), 2.656(85)(multiple), 2.165(46)(multiple), and 1.5589(32)(multiple). The crystal structure was refined to $R_1 = 0.118$ for 2513 observed reflections [$F_o > 4\sigma F$]. The structure contains kinked chains of edge-sharing MnO_6 octahedra parallel to [100]. The chains are linked to each other by corner-sharing, forming sheets parallel to {001} and AsO_4 tetrahedra corner-link with octahedra in the sheet, forming a heteropolyhedral layer. Edge-sharing MnO_6-MnO_5 dimers share corners with octahedra and tetrahedra in adjacent layers, thereby linking them in the [001] direction. The heteropolyhedral layer is topologically identical to those in the structures of the phosphate minerals: angarfitte, bakhchisaraitsevite, mejillonesite, metaswitzerite, rimkorolgitte and switzerite. Overall, the structure is most similar to that of metaswitzerite.</p>
Keywords:	castellaroite; arsenate; crystal structure; Raman spectroscopy; Monte Nero mine; Valletta mine; Italy

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COVER LETTER

Castellaroite, $\text{Mn}^{2+}_3(\text{AsO}_4)_2 \cdot 4.5\text{H}_2\text{O}$, a new mineral from Italy related to metaswitzerite.

Anthony R. Kampf, Fernando Cámara, Marco E. Ciriotti, Barbara P. Nash, Corrado Balestra and
Luigi Chiappino

Herewith, we are submitting a manuscript describing the new mineral castellaroite. The mineral and name have been approved by the Commission on New Minerals, Nomenclature and Classification (IMA).

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COVER PAGE

Title: Castellarroite, $\text{Mn}^{2+}_3(\text{AsO}_4)_2 \cdot 4.5\text{H}_2\text{O}$, a new mineral from Italy related to metaswitzerite.

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Plan of article:

Abstract

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3. Appearance, physical and optical properties

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5. Chemical composition

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7. Discussion of the structure

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References

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Castellaroite, $\text{Mn}^{2+}_3(\text{AsO}_4)_2 \cdot 4.5\text{H}_2\text{O}$, a new mineral from Italy related to metaswitzerite.

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Abstract

Castellaroite (IMA2015-071), $\text{Mn}^{2+}_3(\text{AsO}_4)_2 \cdot 4.5\text{H}_2\text{O}$, is a new secondary arsenate mineral from the Monte Nero mine, Rocchetta Vara, La Spezia, Liguria, Italy and the Valletta mine, near Canosio, Cuneo, Piedmont, Italy. It crystallized from As- and Mn-rich hydrothermal fluids in an oxidizing environment. At Monte Nero, it is associated with coralloite,

manganohörsite, rhodochrosite, sarkinite, sterlinghillite, strashimirite and wallkilldellite. At Valletta, it is associated with braccoite, hematite, manganberzeliite, orthoclase and tiragalloite. Castellarosite occurs as thin blades, flattened on [001], striated and elongated parallel to [100] and exhibiting the forms {110}, {012} and {001}. The mineral is colourless and transparent with a vitreous to silky lustre and white streak. Crystals are flexible with a curved fracture, and one perfect cleavage on {001}. The Mohs' hardness is 2½. The measured and calculated densities are 3.14(2) g·cm⁻³ and 3.164 g·cm⁻³, respectively. The mineral is easily soluble in dilute HCl at room temperature. Optically, castellarosite crystals are biaxial (–), with $\alpha = 1.644(1)$, $\beta = 1.662(1)$ and $\gamma = 1.667(1)$ (white light); $2V = 57(1)^\circ$; dispersion $r < v$, moderate; the optical orientation $Y = \mathbf{b}$; $Z \approx \mathbf{a}$. The Raman spectrum is dominated by features corresponding to the AsO₄ group and also confirms the presence of H₂O. Electron microprobe analyses gave the empirical formula $\text{Mn}^{2+}_{3.02}(\text{As}_{1.94}\text{P}_{0.06})_{\Sigma 2.00}\text{O}_{12.5}\text{H}_{8.96}$, based on 12.5 O *apfu*. Castellarosite is monoclinic, $P2_1/n$, with the unit cell parameters: $a = 8.7565(8)$, $b = 13.4683(13)$, $c = 18.652(2)$ Å, $\beta = 94.876(7)^\circ$, $V = 2191.7(4)$ Å³ and $Z = 8$. The eight strongest lines in the X-ray powder diffraction pattern are [$d_{\text{obs}}/\text{Å}$ (I) (hkl)]: 10.90(100)(011), 9.27(67)(002), 6.97(42)(-111), 3.323(47)(multiple), 3.043(87)(-134,204,-232), 2.656(85)(multiple), 2.165(46)(multiple), and 1.5589(32)(multiple). The crystal structure was refined to $R_1 = 0.118$ for 2513 observed reflections [$F_o > 4\sigma F$]. The structure contains kinked chains of edge-sharing MnO₆ octahedra parallel to [100]. The chains are linked to each other by corner-sharing, forming sheets parallel to {001} and AsO₄ tetrahedra corner-link with octahedra in the sheet, forming a heteropolyhedral layer. Edge-sharing MnO₆–MnO₅ dimers share corners with octahedra and tetrahedra in adjacent layers, thereby linking them in the [001] direction. The heteropolyhedral layer is topologically identical to those in the structures of the phosphate minerals: angarfit, bakhchisaraitsevite, mejillonesite, metaswitzerite, rimkorolgit and switzerite. Overall, the structure is most similar to that of metaswitzerite.

Keywords: castellaroite; arsenate; crystal structure; Raman spectroscopy; Monte Nero mine; Valletta mine; Italy.

1. Introduction

Long inactive manganese deposits, also rich in arsenic, in the Alpine tectono-metamorphic terrains of north-western Italy have yielded a variety of interesting secondary hydrothermal minerals. Among these deposits, the Molinello and Gambatesa mines in Val Graveglia, Liguria are well known, having yielded eight and six new mineral species, respectively. Two lesser-known mines, the Monte Nero mine in Liguria and the Valletta mine in Piedmont have also yielded new species: coralloite (Callegari *et al.*, 2012) from Monte Nero and braccoite (Cámara *et al.*, 2015a), canosioite (Cámara *et al.*, 2015b) and grandaite (Cámara *et al.*, 2014) from Valletta. Herein, we describe the new mineral castellaroite from both the Monte Nero mine and the Valletta mine.

The name castellaroite honours Fabrizio Castellaro (born 1970) of Mezzanego, Italy. Mr. Castellaro is an avid and sophisticated mineral collector specializing in the minerals of Liguria. He is the discoverer of castellaroite and has also discovered the new minerals balestraite, cerchiarait-(Fe) and 1*M* polytype of lavinskyite (“liguriaite”; IMA 2014-035). Mr. Castellaro has agreed to the naming of the mineral in his honour.

The mineral and name (IMA2015–071) were approved by the IMA–CNMNC prior to publication. The description is based upon one holotype and three cotype specimens. The holotype and one cotype, both from the Monte Nero mine, are deposited in the collections of the Natural History Museum of Los Angeles County, 900 Exposition Boulevard, Los Angeles, CA 90007, USA, catalogue numbers 65603 (holotype) and 65604 (cotype). Two cotypes are deposited in the mineralogical collections of the Dipartimento di Scienze della Terra of the

Università di Torino stored in the Museo Regionale di Scienze Naturali di Torino, Sezione di Mineralogia, Petrografia e Geologia, via Giovanni Giolitti 36, I-10123 Torino, Italy, catalogue numbers M/U 16950 (Monte Nero) and M/U 16951 (Valletta).

2. Occurrence

The new mineral was discovered by Fabrizio Castellaro on two specimens collected from the “classic” dump at the Monte Nero mine, Rocchetta Vara, La Spezia, Liguria, Italy (44°14'48"N, 9°45'27"E). The deposit is comprised of thin manganese stratiform ores that are located near the base of a chert sequence in the “Diaspri di Monte Alpe” formation, which overlays Jurassic ophiolites of the Bracco unit. Castellaroite is a secondary mineral that crystallized from As- and Mn-rich fluids, which circulated through a system of fractures during the final tectono-metamorphic stage of the deposit. Other secondary minerals found with castellaroite include coralloite, manganohöernesite, rhodochrosite, sarkinite, sterlinghillite, strashimirite and wallkilldellite. The Monte Nero mine is also the type locality for coralloite (Callegari *et al.*, 2012). More information on the deposit can be found in Callegari *et al.* (2012) and references therein.

Castellaroite has also been found on specimens recovered from the dumps of the Valletta mine, near Canosio, Piedmont, Italy (44°23'42"N, 7°5'42"E), where it was identified by SEM-EDX and Raman spectroscopy, and this is considered to be a cotype locality. The Valletta mine is a small Fe-Mn deposit that has not been worked in modern times and has never been studied geologically or petrologically. The geological setting is rather similar to that of the Monte Nero mine and has been summarized by Cámara *et al.* (2014) in their description of the new mineral grandaite, for which the Valletta mine is also the type locality. At the Valletta mine, castellaroite is a secondary mineral crystallized from hydrothermal

fluids in an oxidizing environment. It is associated with braccoite, hematite, manganberzeliite, orthoclase and tiragalloite.

3. Physical and optical properties

At the Monte Nero mine, castellarosite occurs in radial aggregates up to about 5 mm in diameter consisting of very thin blades, and rarely as individual crystals up to about 2 mm in length (Fig. 1). At the Valletta mine, it forms aggregates of thin blades up to about 0.5 mm in length. Blades are flattened on [001], striated and elongated parallel to [100] and exhibit the forms {110}, {012} and {001} (Fig. 2). No twinning was observed. Crystals are colourless and transparent with a vitreous to silky lustre and white streak. The mineral is non-fluorescent. Crystals are flexible with a curved fracture, and one perfect cleavage on {001}. The Mohs' hardness is 2½, based upon scratch tests. The density measured by floatation in Clerici solution is 3.14(2) g·cm⁻³. The calculated density is 3.164 g·cm⁻³, based on the empirical formula, and 3.174 g·cm⁻³, based on the ideal formula. The mineral is easily soluble in dilute HCl at room temperature. Optically, castellarosite crystals are biaxial (-), with $\alpha = 1.644(1)$, $\beta = 1.662(1)$, $\gamma = 1.667(1)$, measured in white light. The 2V measured directly on a spindle stage is 57(1)°; the calculated 2V is 55°. Dispersion is $r < v$, moderate and the optical orientation is $Y = \mathbf{b}$; $Z \approx \mathbf{a}$. The mineral is nonpleochroic. The Gladstone–Dale compatibility (Mandarino, 2007) is -0.019, in the range of superior compatibility, based upon the empirical formula.

4. Raman spectroscopy

The Raman spectra of castellarosite (Figs. 3 and 4) from both the Monte Nero mine and the Valletta mine were obtained at the Dipartimento di Scienze della Terra (Università di Torino) using a micro/macro Jobin Yvon Mod. LabRam HRVIS, equipped with a motorized x-y stage

and an Olympus microscope. The backscattered Raman signal was collected with 50× objective and the Raman spectra were obtained for a non-oriented crystals. The 532 nm line of a solid state Nd laser was used for excitation; laser power (250 mW) was controlled by means of a series of density filters. The minimum lateral and depth resolution was set to a few μm . The system was calibrated using the 520.6 cm^{-1} Raman band of silicon before each experimental session. The spectra were collected with multiple acquisitions (2 to 3) with single counting times ranging between 20 and 30 seconds. The spectra were recorded using the LabSpec 5 program from 100 to 4000 cm^{-1} . Note that the spectrum obtained for the sample from Valletta is much weaker and, therefore, less well resolved; we only report it here from 100 to 1100 cm^{-1} . Band component analysis was undertaken using the Fityk software package (Wojdyr, 2010).

The Raman spectra confirmed the presence of $(\text{AsO}_4)^{3-}$. The most characteristic features of the castellaroite spectra are observed in the region of $600\text{--}950\text{ cm}^{-1}$ (values observed for Valletta material are reported in italics), where the spectra consist of four main overlapping bands observed at $800.5(800.4)$, $822.2(821.9)$, $846.8(843.5)$ and $862.5(869.9)\text{ cm}^{-1}$, with two weaker shoulders at $910.6(910.2)$ and 933.8 cm^{-1} , the latter not well resolved in the Valletta spectrum (Fig. 3) because it is much weaker. The most intense bands of that group are the one at 862.5 cm^{-1} , which can be interpreted as the ν_1 AsO_4 symmetric stretching vibration, and the one at 800.5 cm^{-1} , which can be interpreted as the ν_3 AsO_4 antisymmetric stretching vibration. The band at $846.8(843.5)$ is not well resolved for the Monte Nero sample and only weakly for the Valletta sample. The weak bands at $910.6(910.2)$ and 933.8 cm^{-1} could be interpreted as water vibrational modes (Martens *et al.*, 2004). At wavenumbers $300\text{--}600\text{ cm}^{-1}$, a prominent band centred at $425.6(426.5)\text{ cm}^{-1}$ is flanked on one side by two well-resolved peaks at $340(346.9)$ and $371.5(376.1)$ and on the other by a peak at $459.4(447.2)\text{ cm}^{-1}$. The bands at 425.6 and 459.4 cm^{-1} can be interpreted as the ν_2 AsO_4 symmetric bending

modes, and the ones at 340 and 371.5 cm^{-1} as the ν_4 AsO_4 antisymmetric bending modes. Two weaker bands observed at 503.6 and 577.5 cm^{-1} are difficult to assign and may be related to the stretching of MnO_6 groups. In the lattice vibrations range ($< 300 \text{ cm}^{-1}$), five weakly resolved bands can be recognized for the Monte Nero sample at 108.9, 149.9, 181.9, 215.1 and 256.9 cm^{-1} , with a shoulder at 143.2 cm^{-1} . The same bands are observed for the Valletta sample at 111.4, 149.6, 177.8, 215.9 and 252.1 cm^{-1} , with the shoulder shifted to lower wavenumbers and better resolved (135.7 cm^{-1}) and a band at 177.8 that is very weak and split into two shoulders of adjacent bands at lower and higher wavenumbers (165.0, 191.4) (Fig. 3). Regarding H_2O group bending and stretching vibrations, in the spectrum for the Monte Nero sample, a very weak band centred at 1662.6 cm^{-1} is observed, which could be interpreted as the bending vibrations of the H_2O . The bands due to hydroxyl stretching modes of the H_2O groups are observed at 2925.1, 3115.9, 3240.9, 3491.4, 3757.7 and 3941.6 cm^{-1} , reflecting the complex nature of the hydrogen bonding on castellaroite (Fig. 4). Note that because of the paucity of material IR spectroscopy was not attempted.

5. Chemical composition

Quantitative analyses (28 points on 7 crystals from Monte Nero) were performed at the University of Utah on a Cameca SX-50 electron microprobe with four wavelength-dispersive spectrometers. Analytical conditions were: 15 kV accelerating voltage, 20 nA beam current and a beam diameter of 10–15 μm . Castellaroite exhibited minor damage under the electron beam. Counting times were 20 seconds on peak and 10 seconds on + and – background. No other elements were detected by EDS. Other likely elements were sought by WDS, but none were above the detection limits. Raw X-ray intensities were corrected for matrix effects with a $\phi(\rho z)$ algorithm (Pouchou and Pichoir, 1991). Because insufficient material was available for a direct determination of H_2O , the amount of water was calculated on the basis of $\text{As} + \text{P} = 2$

apfu, charge balance and $O = 12.5$ *apfu*, as determined by the crystal structure analysis (see below). Analytical data are given in Table 1. The empirical formula is $Mn^{2+}_{3.02}(As_{1.94}P_{0.06})_{\Sigma 2.00}O_{12.5}H_{8.96}$. The simplified structural formula is $Mn^{2+}_3(AsO_4)_2 \cdot 4.5H_2O$, which requires MnO 40.63, As₂O₅ 43.89, H₂O 15.48, total 100 wt%.

5. X-ray crystallography and crystal structure determination

Powder X-ray diffraction data for castellaroite were obtained on a Rigaku R-Axis Rapid II curved-imaging-plate microdiffractometer utilising monochromatised MoK_{α} radiation. A Gandolfi-like motion on the ϕ and ω axes was used to randomize the sample. Observed d spacings and intensities were derived by profile fitting using JADE 2010 software (Materials Data Inc.). Data are given in Table 2. The unit-cell parameters refined from the powder data using JADE 2010 with whole-pattern fitting are: $a = 8.733(4)$, $b = 13.438(4)$, $c = 18.643(4)$ Å, $\beta = 95.006(11)^{\circ}$ and $V = 2179.5(13)$ Å³.

Single-crystal structure data were obtained on the same instrument noted above. The Rigaku CrystalClear software package was used for processing of structure data, including the application of an empirical multi-scan absorption correction using ABSCOR (Higashi, 2001). The structure was solved by direct methods using SIR2011 (Burla *et al.*, 2012). SHELXL-97 (Sheldrick, 2008) was used for the refinement of the structure, with scattering curves for neutral atoms (Wilson, 1992). As and Mn sites were refined with fixed full occupancy. Two O sites in the channel of the structure refined to approximately ½ occupancy and were assigned exactly ½ occupancy in the final refinement. The poor diffraction quality of crystals caused by a strong tendency toward crystal warping resulted in the high R factors and large electron density residuals. In spite of the poor diffraction quality, it was possible to refine anisotropic displacement parameters for all atoms. The very reasonable bond lengths, polyhedral geometries and bond-valence sums corroborate the correctness of the structure. Data

collection and refinement details are given in Table 3, atom coordinates and displacement parameters in Table 4, selected bond distances in Table 5 and a bond valence analysis in Table 6. The Crystallography Information File (CIF), including reflection data, is available online as supplementary material linked to this article on the GSW website of the journal, <http://eurjmin.geoscience.world.org>

6. Discussion of the structure

In the structure of castellaroite, there are four different AsO_4 tetrahedra, five different MnO_6 octahedra and one MnO_5 square pyramid. The octahedra centred by Mn1, Mn2, Mn3 and Mn4 share edges forming kinked chains parallel to $[100]$. The chains are linked to each other by corner-sharing, forming sheets parallel to $\{001\}$. All four AsO_4 tetrahedra corner-link with octahedra in the sheet, forming a heteropolyhedral layer (Fig. 5). Mn5O_6 – Mn6O_5 edge-sharing dimers are located between the heteropolyhedral layers and share corners with octahedra and tetrahedra in adjacent layers, thereby linking the layers in the $[001]$ direction.

The heteropolyhedral layer is topologically identical to those in the structures of several phosphate minerals: angarfite, $\text{NaFe}^{3+}_5(\text{PO}_4)_4(\text{OH})_4 \cdot 4\text{H}_2\text{O}$ (Kampf *et al.*, 2012), bakhchisaraitsevite, $\text{Na}_2\text{Mg}_5(\text{PO}_4)_4 \cdot 7\text{H}_2\text{O}$ (Liferovich *et al.*, 2000; Yakubovich *et al.*, 2000), mejillonesite, $\text{NaMg}_2(\text{PO}_3\text{OH})(\text{PO}_4)(\text{OH}) \cdot (\text{H}_3\text{O} + \text{H}_2\text{O})$ (Atencio *et al.*, 2012), metaswitzerite, $\text{Mn}_3(\text{PO}_4)_2 \cdot 4\text{H}_2\text{O}$ (formerly switzerite; Fanfani and Zanazzi, 1979), rimkorolgit, $\text{BaMg}_5(\text{PO}_4)_4 \cdot 8\text{H}_2\text{O}$ (Krivovichev *et al.*, 2002) and switzerite, $\text{Mn}_3(\text{PO}_4)_2 \cdot 7\text{H}_2\text{O}$ (Zanazzi *et al.*, 1986). Castellaroite is the first arsenate with this type of heteropolyhedral layer. The interlayer region in the structure of castellaroite is different from that in any of these structures, but it is most similar to that in metaswitzerite, which except for the configuration of Mn5O_6 and Mn6O_5 polyhedra, could be regarded as the PO_4 analogue of castellaroite.

In the metaswitzerite structure, pairs of Mn_6O_5 pyramids form edge-sharing dimers and Mn_5O_6 octahedra do not share edges with any other polyhedra. Although the linkages in both structures result in heteropolyhedral frameworks, the castellarite framework contains continuous channels along [100], while the metaswitzerite framework contains discontinuous cavities. Isolated H_2O groups are contained in the channels in castellarite (O24, O25 and O26) and the cavities in metaswitzerite (O24). The structures of castellarite and metaswitzerite are compared in Figure 6.

The layers in the structure of switzerite, including outlying MnO_6 octahedra, are linked to adjacent layers only by hydrogen bonds through isolated H_2O groups in the interlayer region. Switzerite transforms readily to metaswitzerite by losing $\frac{3}{4}$ of its isolated H_2O groups resulting in the condensation of the layers into the framework noted above (Zanazzi *et al.*, 1986). An arsenate equivalent of switzerite, which could correspond to a hydrated precursor to castellarite, is not known. Manganohörsesite, $\text{Mn}_3(\text{AsO}_4)_2 \cdot 8\text{H}_2\text{O}$, a member of the vivianite group, has a structure very different from that of switzerite and cannot transition to castellarite through dehydration via simple structural condensation.

It is worth noting that sterlinghillite, $\text{Mn}_3(\text{AsO}_4)_2 \cdot 3\text{H}_2\text{O}$ (Dunn, 1981; Matsubara *et al.*, 2000), which has been found with castellarite, has $1\frac{1}{2}$ less H_2O groups *pfu*. Its structure has never been determined; however, its powder XRD is quite distinct from that of castellarite. The sterlinghillite from the Monte Nero mine that we examined occurs as subparallel intergrowths of tiny plates. The plates are distinctly brittle and much harder than castellarite. Unfortunately, efforts to separate a plate suitable for single-crystal study were unsuccessful.

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 348

Figure 1



Figure 1. Castellarosite crystals from the Monte Nero mine. FOV 4 mm across.

Figure 2

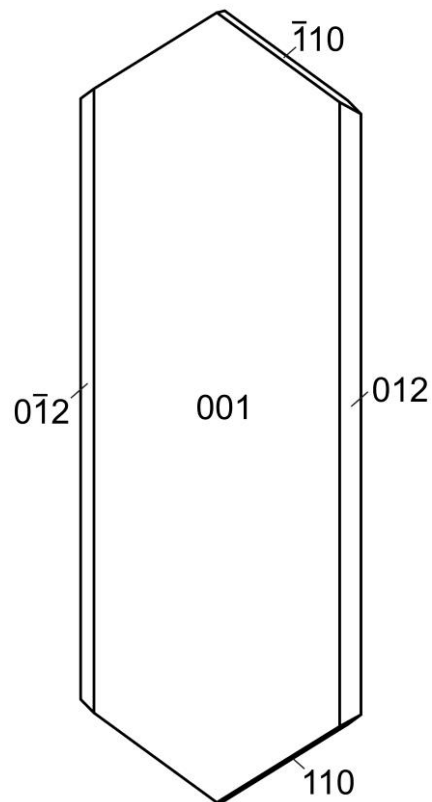


Figure 2. Crystal drawing of castellarosite, clinographic projection in nonstandard orientation, $[100]$ vertical.

Figure 3

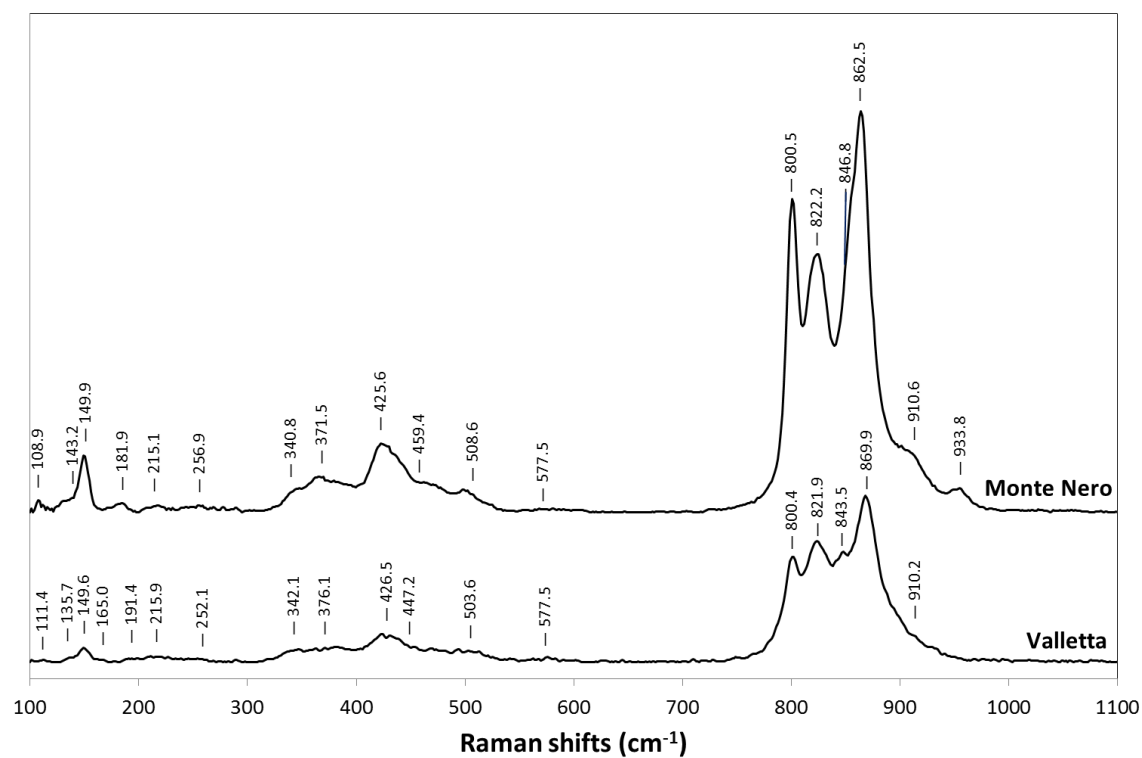


Figure 3. Raman spectra of castellaroite from the Monte Nero and Valletta mines in the 100–1100 cm⁻¹ range.

Figure 4

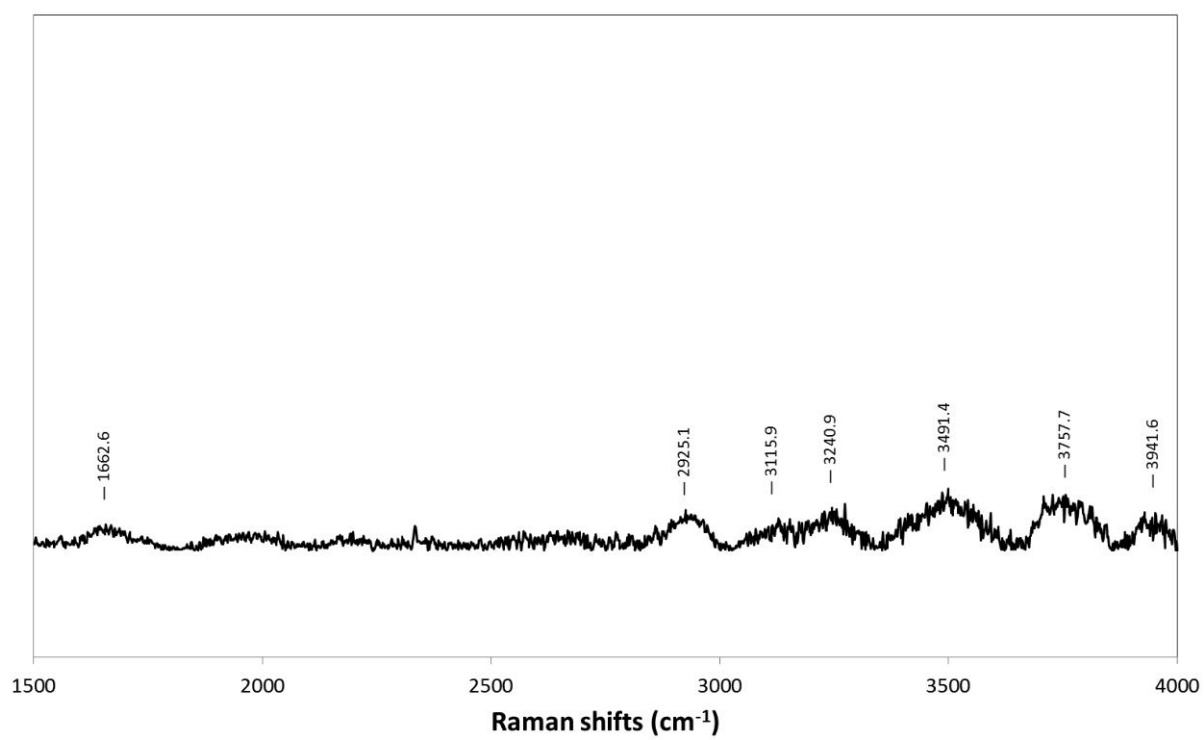


Figure 4. Raman spectrum of castellaroite from the Monte Nero mine in the 1500–4000 cm⁻¹ range.

Figure 5

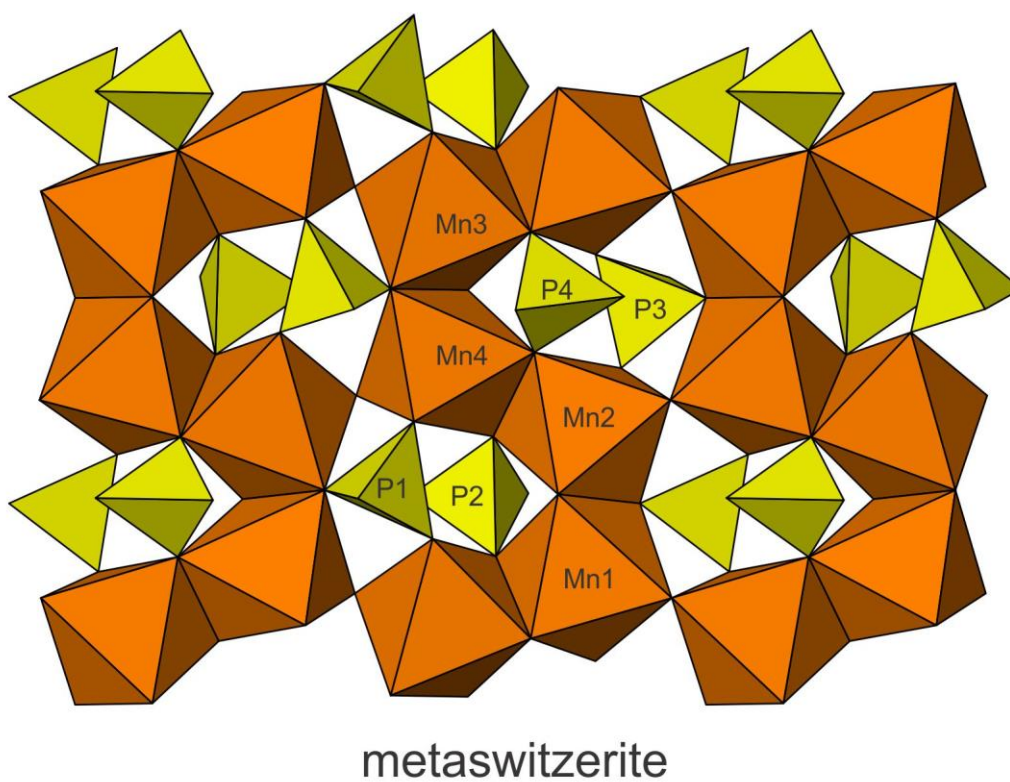
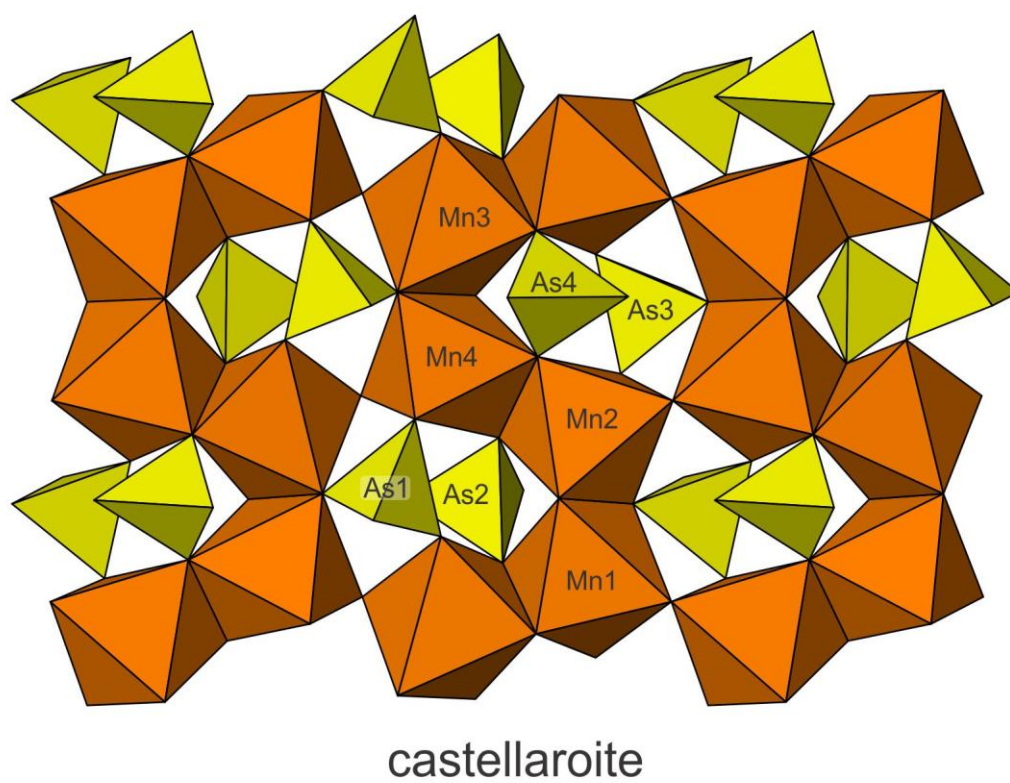


Figure 5. Polyhedral layers parallel to {001} in castellaroite and metaswitzerite, viewed with [100] vertical.

Figure 6

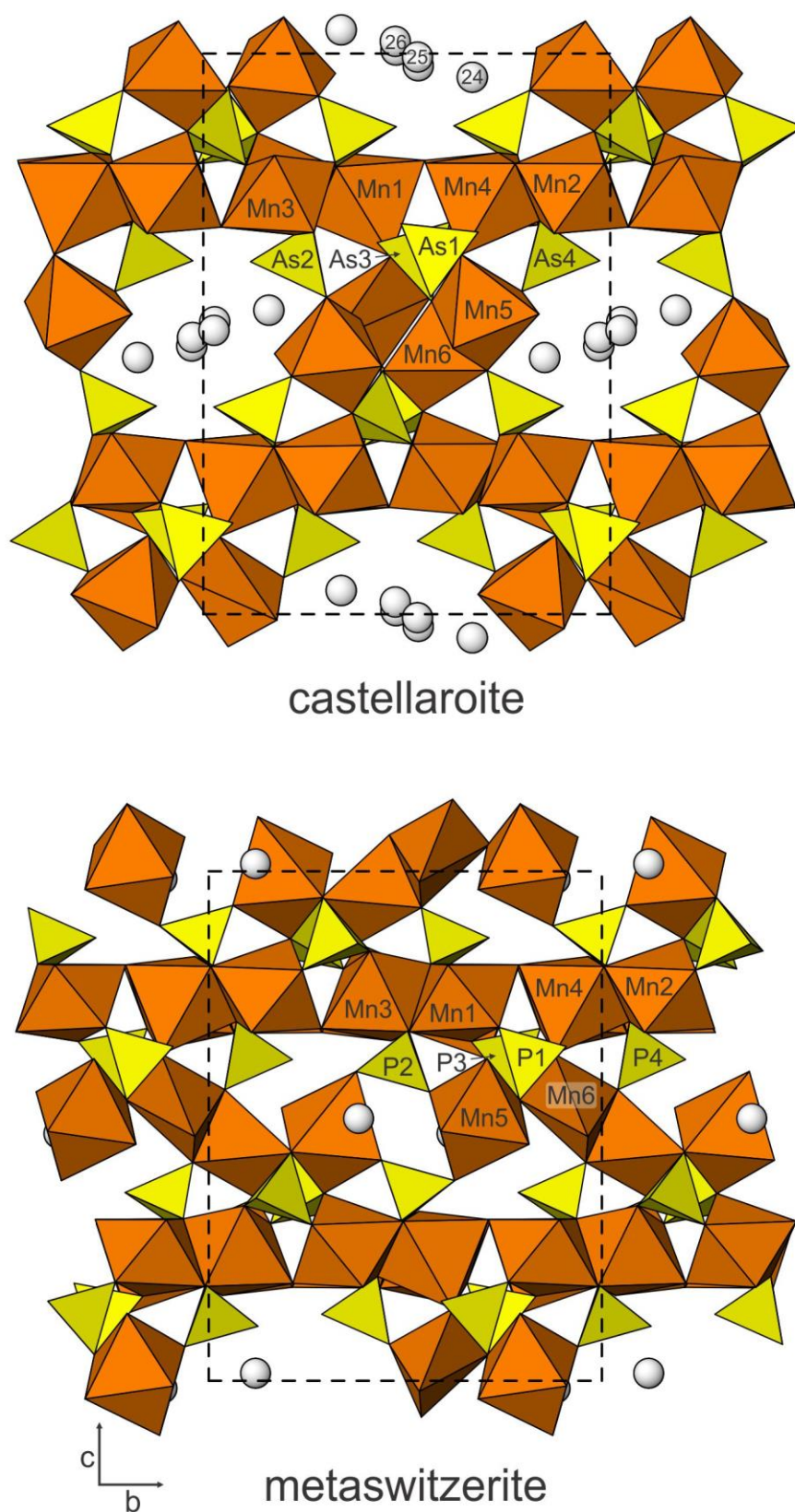


Figure 6. The crystal structures of castellaroite and metaswitzerite viewed parallel to {100}. The channel H₂O groups are shown as white spheres and are numbered for the castellaroite structure. The unit cells are shown by dashed lines.

Table 1. Analytical data (in wt%) for castellaroite.

Constituent	Mean	Range	SD	Standard
MnO	41.62	40.33–42.82	0.63	rhodonite
As ₂ O ₅	43.35	41.83–44.64	0.75	syn. GaAs
P ₂ O ₅	0.82	0.61–1.23	0.16	apatite
H ₂ O*	15.69			
Total	101.48			

* Calculated on the basis of As+P = 2 *apfu*, charge balance and O = 12.5 *apfu*.

Table 2. Powder X-ray data (d in Å) for castellaroite. Only calculated lines with $I > 1$ are shown.

I_{obs}	d_{obs}	d_{calc}	I_{calc}	hkl	I_{obs}	d_{obs}	d_{calc}	I_{calc}	hkl
100	10.90	10.9056	100	0 1 1			2.1812	4	4 0 0
67	9.27	9.2922	84	0 0 2			2.1726	6	3 1 5
		7.6485	4	0 1 2	46	2.165	2.1648	10	-4 0 2
42	6.97	6.9849	28	-1 1 1			2.1539	6	1 6 1
26	6.63	6.6529	22	1 1 1			2.0925	2	3 2 5
		5.6280	3	0 1 3	7	2.088	2.0848	2	0 4 7
		5.5618	5	1 1 2			2.0632	2	0 3 8
11	5.20	5.1963	7	-1 2 1			2.0424	2	-4 0 4
		4.7313	5	-1 2 2	10	2.0355	2.0378	2	1 6 3
9	4.609	4.5714	10	1 1 3			2.0261	3	-3 1 7
10	4.373	4.3624	7	2 0 0			1.9660	2	2 3 7
		4.1487	3	-1 2 3	7	1.9505	1.9357	2	4 3 1
12	4.031	4.0424	8	0 3 2	6	1.9218	1.9129	2	4 0 4
		3.9920	2	1 3 0	9	1.8951	1.8906	3	4 3 2
		3.8721	2	1 3 1			1.8646	2	3 1 7
11	3.830	3.8258	6	2 0 2	18	1.8590	1.8593	2	-4 0 6
17	3.692	3.7207	8	-1 3 2			1.8585	6	0 0 10
		3.6352	11	0 3 3			1.8576	3	-3 5 4
		3.6172	3	1 3 2			1.7877	2	-2 6 5
24	3.594	3.5963	22	-1 2 4			1.7603	4	0 4 9
9	3.453	3.4158	18	1 2 4	6	1.7504	1.7464	3	-1 5 8
47	3.323	3.3244	22	-2 0 4			1.7225	3	4 4 3
		3.3131	3	0 4 1	9	1.7167	1.6988	2	-3 2 9
		3.2285	6	0 3 4			1.6885	2	3 2 8
		3.2229	2	1 1 5	13	1.6830	1.6851	5	3 6 3
20	3.115	3.1286	7	2 3 0			1.6764	2	0 1 11
		3.1261	12	-1 2 5			1.6384	2	5 2 2
		3.0878	3	-1 3 4	18	1.6295	1.6366	7	-3 6 5
87	3.043	3.0534	50	2 0 4			1.6312	2	-5 2 4
		3.0213	15	-2 3 2			1.6240	4	3 5 6
17	2.961	2.9773	6	1 2 5			1.5967	2	5 2 3
		2.9583	5	0 4 3			1.5928	3	2 4 9
		2.9119	9	2 3 2	20	1.5819	1.5844	4	-3 2 10
18	2.894	2.8872	3	-2 1 5			1.5828	3	0 8 4
		2.8638	8	-2 3 3			1.5750	2	3 2 9
		2.7407	6	-1 2 6			1.5717	4	3 6 5
		2.7264	4	0 4 4			1.5706	2	2 8 0
		2.6723	8	-3 2 1			1.5691	3	-2 8 1
85	2.656	2.6699	12	3 2 0	32	1.5589	1.5600	2	-4 4 7
		2.6638	5	2 1 5			1.5588	2	-4 3 8
		2.6579	18	-2 4 1			1.5565	4	-2 8 2
		2.6334	18	-2 0 6			1.5472	3	5 2 4
17	2.614	2.6197	7	1 2 6			1.5371	4	-5 2 6
8	2.566	2.5872	6	0 5 2	15	1.5290	1.5336	3	0 8 5
		2.5495	8	0 3 6			1.5133	4	-1 6 9
15	2.492	2.4954	13	0 4 5	6	1.4760	1.4816	3	1 6 9
		2.4646	3	1 5 2			1.4617	2	-2 4 11
3	2.418	2.4033	4	-3 3 2	19	1.4550	1.4557	2	-6 0 2
4	2.327	2.3303	4	0 5 4			1.4395	2	-1 9 3
		2.3231	2	0 0 8	10	1.4296	1.4229	2	-2 3 12
		2.2852	2	0 3 7	12	1.3783	1.3807	4	-5 6 1
7	2.216	2.2285	2	0 6 1					
		2.2214	3	-2 4 5					

Table 3. Data collection and structure refinement details for castellaroite.

Diffractometer	Rigaku R-Axis Rapid II
X-ray radiation / power	MoK α ($\lambda = 0.71075$ Å)/50 kV, 40 mA
Temperature	293(2) K
Structural Formula	Mn ₃ (AsO ₄) ₂ ·4.5H ₂ O
Space group	<i>P</i> 2 ₁ / <i>n</i>
Unit cell dimensions	<i>a</i> = 8.7565(8) Å <i>b</i> = 13.4683(13) Å <i>c</i> = 18.652(2) Å β = 94.876(7)°
<i>V</i>	2191.7(4) Å ³
<i>Z</i>	8
Density (for above formula)	3.174 g cm ⁻³
Absorption coefficient	9.458 mm ⁻¹
<i>F</i> (000)	2000
Crystal size	240 × 45 × 5 μm
θ range	3.03 to 25.25°
Index ranges	$-10 \leq h \leq 10$, $-15 \leq k \leq 15$, $-22 \leq l \leq 22$
Refls. collected / unique	17418 / 3407; $R_{\text{int}} = 0.093$
Reflections with $F > 4\sigma(F)$	2513
Completeness to $\theta = 19.96^\circ$	85.9%
Max. and min. transmission	0.954 and 0.210
Refinement method	Full-matrix least-squares on F^2
Parameters / restraints	325 / 0
GoF	1.346
Final <i>R</i> indices [$F_o > 4\sigma(F)$]	$R_1 = 0.1182$, $wR_2 = 0.3270$
<i>R</i> indices (all data)	$R_1 = 0.1365$, $wR_2 = 0.3503$
Largest diff. peak / hole	+4.87 / -3.68 e/Å ³

* $R_{\text{int}} = \Sigma|F_o^2 - F_o^2(\text{mean})|/\Sigma[F_o^2]$. GoF = $S = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n-p)\}^{1/2}$. $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$; $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where *a* is 0.2, *b* is 0 and *P* is $[2F_c^2 + \text{Max}(F_o^2, 0)]/3$.

Table 4

Table 4. Atom coordinates and displacement parameters (\AA^2) for castellaroite.

	x/a	y/b	z/c	U_{eq}	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Mn1	0.9614(3)	0.58628(18)	0.26437(14)	0.0238(7)	0.0210(15)	0.0110(14)	0.0400(17)	0.0017(9)	0.0063(10)	-0.0004(10)
Mn2	0.5628(3)	0.58800(18)	0.26092(14)	0.0233(7)	0.0200(14)	0.0149(15)	0.0349(16)	0.0021(9)	0.0018(10)	-0.0001(10)
Mn3	0.4562(3)	0.31797(18)	0.25484(14)	0.0235(7)	0.0208(14)	0.0109(15)	0.0395(17)	-0.0016(9)	0.0061(10)	-0.0001(10)
Mn4	0.0640(3)	0.31829(17)	0.25889(14)	0.0232(7)	0.0177(14)	0.0118(15)	0.0401(17)	-0.0010(9)	0.0027(10)	-0.0029(10)
Mn5	0.1037(3)	0.31049(19)	0.45711(15)	0.0275(7)	0.0219(15)	0.0221(16)	0.0384(17)	-0.0039(10)	0.0021(11)	-0.0003(10)
Mn6	0.3812(3)	0.42197(18)	0.54981(14)	0.0267(7)	0.0263(15)	0.0162(15)	0.0373(17)	0.0000(10)	0.0009(10)	-0.0040(10)
As1	0.21688(18)	0.58951(13)	0.64641(9)	0.0228(6)	0.0208(11)	0.0128(11)	0.0353(12)	0.0004(6)	0.0048(7)	-0.0006(6)
As2	0.23328(19)	0.23738(13)	0.64198(9)	0.0244(6)	0.0211(11)	0.0162(11)	0.0363(12)	0.0015(6)	0.0046(7)	-0.0005(7)
As3	0.28564(18)	0.48655(12)	0.36085(9)	0.0215(6)	0.0183(10)	0.0115(11)	0.0350(12)	-0.0011(6)	0.0033(7)	-0.0014(6)
As4	0.27166(18)	0.14694(14)	0.35133(9)	0.0245(6)	0.0208(11)	0.0158(11)	0.0372(12)	-0.0007(6)	0.0052(7)	0.0007(7)
O1	0.6658(13)	0.3246(8)	0.3185(7)	0.031(3)	0.026(6)	0.016(6)	0.050(8)	-0.002(5)	-0.008(5)	-0.006(5)
O2	0.2718(12)	0.5586(8)	0.5659(6)	0.032(3)	0.020(6)	0.021(7)	0.056(9)	-0.001(5)	0.014(5)	0.012(5)
O3	0.7709(12)	0.5142(8)	0.3035(6)	0.026(3)	0.019(6)	0.008(6)	0.051(8)	0.007(4)	0.003(5)	0.005(5)
O4	0.9644(11)	0.3663(8)	0.3585(6)	0.023(2)	0.020(6)	0.016(6)	0.033(7)	-0.007(4)	0.000(4)	0.002(4)
O5	0.2281(12)	0.1159(9)	0.6288(6)	0.029(3)	0.024(6)	0.028(7)	0.036(7)	-0.006(5)	0.006(5)	0.006(5)
O6	0.5765(12)	0.2280(8)	0.1810(6)	0.026(3)	0.020(6)	0.014(6)	0.045(8)	-0.006(4)	0.012(5)	-0.002(5)
O7	0.2409(13)	0.3026(8)	0.5653(6)	0.029(3)	0.033(7)	0.026(7)	0.027(7)	-0.009(5)	0.001(5)	0.001(5)
O8	0.8912(11)	0.2335(8)	0.1962(6)	0.025(3)	0.009(5)	0.018(6)	0.048(8)	-0.009(5)	0.007(4)	-0.004(4)
O9	0.1599(13)	0.5728(8)	0.3372(6)	0.028(3)	0.030(7)	0.011(6)	0.043(8)	-0.004(5)	-0.004(5)	-0.003(5)
O10	0.2727(13)	0.3943(8)	0.3008(6)	0.030(3)	0.029(7)	0.029(7)	0.033(7)	0.001(5)	0.004(5)	-0.005(5)
O11	0.2603(12)	0.4389(9)	0.4422(6)	0.027(3)	0.025(6)	0.039(8)	0.020(6)	0.000(5)	0.010(4)	-0.006(5)
O12	0.4622(12)	0.5394(8)	0.3616(6)	0.027(3)	0.031(6)	0.017(6)	0.031(7)	0.001(4)	0.000(5)	-0.005(5)
O13	0.2772(12)	0.2173(8)	0.4243(6)	0.031(3)	0.022(6)	0.027(7)	0.045(8)	-0.005(5)	0.008(5)	-0.005(5)
O14	0.2710(13)	0.0260(8)	0.3696(6)	0.027(3)	0.033(7)	0.017(6)	0.031(7)	0.003(4)	0.003(5)	0.001(5)
O15	0.4225(11)	0.1762(8)	0.3056(6)	0.024(2)	0.014(5)	0.019(7)	0.040(7)	-0.004(4)	0.009(4)	-0.005(5)
O16	0.1098(12)	0.1733(8)	0.2983(6)	0.028(3)	0.020(6)	0.029(7)	0.034(7)	-0.003(5)	-0.010(4)	-0.002(5)
OW17	0.4943(12)	0.4560(8)	0.1905(6)	0.027(3)	0.023(6)	0.015(6)	0.042(7)	-0.002(4)	0.000(5)	0.004(5)
OW18	0.2432(12)	0.2744(8)	0.1769(6)	0.026(3)	0.025(6)	0.013(6)	0.040(7)	-0.010(4)	0.000(5)	0.011(5)
OW19	0.9946(12)	0.4539(8)	0.1954(6)	0.027(3)	0.024(6)	0.003(6)	0.053(8)	0.005(4)	0.006(5)	-0.005(5)

OW20	0.7623(12)	0.1379(8)	0.3212(7)	0.032(3)	0.026(7)	0.014(7)	0.058(9)	-0.006(5)	0.013(5)	0.006(5)
OW21	0.9554(16)	0.3867(13)	0.5258(8)	0.066(5)	0.056(10)	0.100(14)	0.042(9)	-0.014(8)	0.001(7)	0.038(9)
OW22	0.9491(16)	0.1751(10)	0.4646(9)	0.058(4)	0.049(9)	0.037(9)	0.089(13)	0.006(7)	0.013(7)	-0.001(7)
OW23	0.5233(13)	0.3192(9)	0.4894(7)	0.037(3)	0.025(7)	0.024(8)	0.065(10)	-0.003(5)	0.010(6)	0.000(5)
OW24	0.1836(18)	0.3389(11)	0.0422(8)	0.064(4)	0.086(12)	0.046(10)	0.061(11)	0.012(7)	0.000(8)	-0.007(8)
OW25*	0.127(3)	0.0263(19)	0.5063(12)	0.043(6)	0.067(18)	0.038(16)	0.023(14)	0.006(10)	0.005(11)	-0.003(13)
OW26*	0.379(4)	0.028(2)	0.5217(16)	0.066(9)	0.09(2)	0.05(2)	0.06(2)	-0.008(14)	0.019(17)	-0.020(17)

* OW25 and OW26 are half occupied.

Table 5. Selected bond distances (Å) for castellaroite.

Mn1–O15	2.105(11)	Mn4–O16	2.113(11)	As1–O1	1.646(11)
Mn1–O3	2.113(11)	Mn4–O8	2.158(10)	As1–O2	1.668(11)
Mn1–O9	2.121(11)	Mn4–O10	2.181(11)	As1–O3	1.679(10)
Mn1–O6	2.202(11)	Mn4–O4	2.215(10)	As1–O4	1.690(10)
Mn1–OW19	2.232(11)	Mn4–OW19	2.233(11)	< As1–O>	1.671
Mn1–OW20	2.517(12)	Mn4–OW18	2.357(11)		
< Mn1–O>	2.215	< Mn4–O>	2.210	As2–O5	1.655(12)
				As2–O6	1.674(10)
Mn2–O16	2.131(10)	Mn5–O13	2.101(11)	As2–O7	1.684(12)
Mn2–O8	2.142(11)	Mn5–OW21	2.159(14)	As2–O8	1.689(11)
Mn2–O3	2.166(10)	Mn5–O11	2.239(11)	< As2–O>	1.676
Mn2–O12	2.238(11)	Mn5–O4	2.249(11)		
Mn2–OW17	2.261(11)	Mn5–O7	2.262(12)	As3–O9	1.635(11)
Mn2–OW20	2.354(11)	Mn5–OW22	2.282(14)	As3–O10	1.670(11)
< Mn2–O>	2.215	< Mn5–O>	2.215	As3–O11	1.679(11)
				As3–O12	1.701(10)
Mn3–O1	2.101(11)	Mn6–O7	2.059(12)	< As3–O>	1.671
Mn3–O10	2.147(12)	Mn6–O2	2.107(11)		
Mn3–O15	2.163(11)	Mn6–O12	2.121(11)	As4–O13	1.656(11)
Mn3–O6	2.173(10)	Mn6–O11	2.200(11)	As4–O14	1.664(11)
Mn3–OW17	2.253(11)	Mn6–OW23	2.230(13)	As4–O15	1.679(10)
Mn3–OW18	2.340(11)	< Mn6–O>	2.143	As4–O16	1.695(10)
< Mn3–O>	2.196			< As4–O>	1.674
Hydrogen bonds					
OW17–O5	2.619(16)	OW20–O1	2.651(15)	OW23–O2	2.704(17)
OW17–O14	2.664(15)	OW20–O15	3.009(15)	OW23–O13	2.749(16)
OW18–OW24	2.667(18)	OW21–O2	2.617(18)	OW24–OW23	2.69(2)
OW18–O9	2.864(14)	OW21–O9	2.878(18)	OW24–O14	3.018(19)
OW19–O14	2.655(15)	OW22–OW24	2.85(2)		
OW19–O5	2.716(15)	OW22–OW20	3.06(2)		

Table 6. Bond-valence analysis for castellarosite. Values are expressed in valence units.

	Mn1	Mn2	Mn3	Mn4	Mn5	Mn6	As1	As2	As3	As4	hydrogen bonds	Σ_a
O1			0.43				1.39				+0.25	2.07
O2						0.42	1.31				+0.28,+0.22	2.23
O3	0.42	0.36					1.27					2.05
O4				0.32	0.29		1.23					1.84
O5								1.35			+0.27,+0.22	1.84
O6	0.33		0.36					1.29				1.98
O7					0.28	0.48		1.25				2.01
O8		0.39		0.37				1.23				1.99
O9	0.41								1.43		+0.16,+0.16	2.16
O10			0.38	0.35					1.30			2.03
O11					0.30	0.33			1.27			1.90
O12		0.30				0.41			1.20			1.91
O13					0.43					1.35	+0.20	1.98
O14										1.32	+0.25,+0.25,+0.13	1.95
O15	0.43		0.36							1.27	+0.13	2.19
O16		0.40		0.42						1.21		2.03
OW17		0.28	0.29								−0.25,−0.27	0.05
OW18			0.23	0.22							−0.16,−0.24	0.05
OW19	0.30			0.30							−0.22,−0.25	0.13
OW20	0.14	0.22									+0.12,−0.13,−0.25	0.10
OW21					0.37						−0.16,−0.28	−0.07
OW22					0.26						−0.12,−0.17	−0.03
OW23						0.30					+0.23,−0.20,−0.22	0.11
OW24											+0.17,+0.24,−0.13,−0.23	0.05
Σ_c	2.16	1.97	2.02	1.96	1.94	1.90	5.13	4.94	5.32	5.01		

Bond valence parameters from Brown and Altermatt (1985). Hydrogen-bond strengths based on O–O bond distances from Ferraris, G. and Ivaldi, G. (1988). Half-occupied OW25 and OW26 sites are not included. Donated and accepted hydrogen bonds are indicated by – and +, respectively.

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Mn4 Mn 0.0640(3) 0.31829(17) 0.25889(14) 0.0232(7) Uani 1 1 d . . . . .
Mn5 Mn 0.1037(3) 0.31049(19) 0.45711(15) 0.0275(7) Uani 1 1 d . . . . .
Mn6 Mn 0.3812(3) 0.42197(18) 0.54981(14) 0.0267(7) Uani 1 1 d . . . . .
As1 As 0.21688(18) 0.58951(13) 0.64641(9) 0.0228(6) Uani 1 1 d . . . . .
As2 As 0.23328(19) 0.23738(13) 0.64198(9) 0.0244(6) Uani 1 1 d . . . . .
As3 As 0.28564(18) 0.48655(12) 0.36085(9) 0.0215(6) Uani 1 1 d . . . . .
As4 As 0.27166(18) 0.14694(14) 0.35133(9) 0.0245(6) Uani 1 1 d . . . . .
O1 O 0.6658(13) 0.3246(8) 0.3185(7) 0.031(3) Uani 1 1 d . . . . .
O2 O 0.2718(12) 0.5586(8) 0.5659(6) 0.032(3) Uani 1 1 d . . . . .
O3 O 0.7709(12) 0.5142(8) 0.3035(6) 0.026(3) Uani 1 1 d . . . . .
O4 O 0.9644(11) 0.3663(8) 0.3585(6) 0.023(2) Uani 1 1 d . . . . .
O5 O 0.2281(12) 0.1159(9) 0.6288(6) 0.029(3) Uani 1 1 d . . . . .

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O7	O	0.2409(13)	0.3026(8)	0.5653(6)	0.029(3)	Uani	1	1	d
O8	O	0.8912(11)	0.2335(8)	0.1962(6)	0.025(3)	Uani	1	1	d
O9	O	0.1599(13)	0.5728(8)	0.3372(6)	0.028(3)	Uani	1	1	d
O10	O	0.2727(13)	0.3943(8)	0.3008(6)	0.030(3)	Uani	1	1	d
O11	O	0.2603(12)	0.4389(9)	0.4422(6)	0.027(3)	Uani	1	1	d
O12	O	0.4622(12)	0.5394(8)	0.3616(6)	0.027(3)	Uani	1	1	d
O13	O	0.2772(12)	0.2173(8)	0.4243(6)	0.031(3)	Uani	1	1	d
O14	O	0.2710(13)	0.0260(8)	0.3696(6)	0.027(3)	Uani	1	1	d
O15	O	0.4225(11)	0.1762(8)	0.3056(6)	0.024(2)	Uani	1	1	d
O16	O	0.1098(12)	0.1733(8)	0.2983(6)	0.028(3)	Uani	1	1	d
OW17	O	0.4943(12)	0.4560(8)	0.1905(6)	0.027(3)	Uani	1	1	d
OW18	O	0.2432(12)	0.2744(8)	0.1769(6)	0.026(3)	Uani	1	1	d
OW19	O	0.9946(12)	0.4539(8)	0.1954(6)	0.027(3)	Uani	1	1	d
OW20	O	0.7623(12)	0.1379(8)	0.3212(7)	0.032(3)	Uani	1	1	d
OW21	O	0.9554(16)	0.3867(13)	0.5258(8)	0.066(5)	Uani	1	1	d
OW22	O	0.9491(16)	0.1751(10)	0.4646(9)	0.058(4)	Uani	1	1	d
OW23	O	0.5233(13)	0.3192(9)	0.4894(7)	0.037(3)	Uani	1	1	d
OW24	O	0.1836(18)	0.3389(11)	0.0422(8)	0.064(4)	Uani	1	1	d
OW25	O	0.127(3)	0.0263(19)	0.5063(12)	0.043(6)	Uani	0.5	1	d	.	.	.	P	.	.
OW26	O	0.379(4)	0.028(2)	0.5217(16)	0.066(9)	Uani	0.5	1	d	.	.	.	P	.	.

loop_

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Mn2	0.0200(14)	0.0149(15)	0.0349(16)	0.0021(9)	0.0018(10)	-0.0001(10)	
Mn3	0.0208(14)	0.0109(15)	0.0395(17)	-0.0016(9)	0.0061(10)	-0.0001(10)	
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Mn5	0.0219(15)	0.0221(16)	0.0384(17)	-0.0039(10)	0.0021(11)	-0.0003(10)	
Mn6	0.0263(15)	0.0162(15)	0.0373(17)	0.0000(10)	0.0009(10)	-0.0040(10)	
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As2	0.0211(11)	0.0162(11)	0.0363(12)	0.0015(6)	0.0046(7)	-0.0005(7)	
As3	0.0183(10)	0.0115(11)	0.0350(12)	-0.0011(6)	0.0033(7)	-0.0014(6)	
As4	0.0208(11)	0.0158(11)	0.0372(12)	-0.0007(6)	0.0052(7)	0.0007(7)	
O1	0.026(6)	0.016(6)	0.050(8)	-0.002(5)	-0.008(5)	-0.006(5)	
O2	0.020(6)	0.021(7)	0.056(9)	-0.001(5)	0.014(5)	0.012(5)	
O3	0.019(6)	0.008(6)	0.051(8)	0.007(4)	0.003(5)	0.005(5)	
O4	0.020(6)	0.016(6)	0.033(7)	-0.007(4)	0.000(4)	0.002(4)	
O5	0.024(6)	0.028(7)	0.036(7)	-0.006(5)	0.006(5)	0.006(5)	
O6	0.020(6)	0.014(6)	0.045(8)	-0.006(4)	0.012(5)	-0.002(5)	
O7	0.033(7)	0.026(7)	0.027(7)	-0.009(5)	0.001(5)	0.001(5)	
O8	0.009(5)	0.018(6)	0.048(8)	-0.009(5)	0.007(4)	-0.004(4)	
O9	0.030(7)	0.011(6)	0.043(8)	-0.004(5)	-0.004(5)	-0.003(5)	
O10	0.029(7)	0.029(7)	0.033(7)	0.001(5)	0.004(5)	-0.005(5)	
O11	0.025(6)	0.039(8)	0.020(6)	0.000(5)	0.010(4)	-0.006(5)	
O12	0.031(6)	0.017(6)	0.031(7)	0.001(4)	0.000(5)	-0.005(5)	
O13	0.022(6)	0.027(7)	0.045(8)	-0.005(5)	0.008(5)	-0.005(5)	
O14	0.033(7)	0.017(6)	0.031(7)	0.003(4)	0.003(5)	0.001(5)	

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OW18 0.025(6) 0.013(6) 0.040(7) -0.010(4) 0.000(5) 0.011(5)
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OW25 0.067(18) 0.038(16) 0.023(14) 0.006(10) 0.005(11) -0.003(13)
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Mn4 O8 2.158(10) 1_455 ?

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_shelxl_version_number 2013-4

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TITL CL179

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ZERR 8 0.00076 0.00133 0.00240 0.00000 0.00670 0.00000

LATT 1

SYMM 1/2-X, 1/2+Y, 1/2-Z

SFAC AS H MN O

UNIT 16 72 24 100

SHEL 99999 0.833

L.S. 8

omit 3 6 2

omit -9 6 4

omit 5 6 0

EQIV \$1 -x+1/2, y+1/2, -z+1/2

EQIV \$2 x+1/2, -y+1/2, z-1/2

EQIV \$3 -x+1/2, y-1/2, -z+1/2

EQIV \$4 -x+3/2, y+1/2, -z+1/2

EQIV \$5 x+1/2, -y+1/2, z+1/2

EQIV \$6 -x+1, -y+1, -z+1

EQIV \$7 x-1/2, -y+1/2, z-1/2

BIND OW17 O14_\$1

BIND OW17 O5_\$2

BIND OW18 O9_\$3

BIND OW18 OW24

BIND OW19 O14_\$4

BIND OW19 O5_\$2

BIND OW20 O15

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BIND OW20 O1
BIND OW21 O9_$6
BIND OW21 O2_$6
BIND OW22 OW20
BIND OW22 OW24_$5
BIND OW23 O13
BIND OW23 O2_$6
BIND OW24 O14_$1
BIND OW24 OW23_$7
FMAP 2
PLAN 8
ACTA
SIZE 0.240 0.045 0.005
WGHT 0.200000
FVAR 0.28146
MN1 3 0.961380 0.586277 0.264369 11.00000 0.02105
0.01104 =
0.03997 0.00172 0.00631 -0.00039
MN2 3 0.562754 0.588001 0.260920 11.00000 0.02000
0.01490 =
0.03494 0.00206 0.00177 -0.00011
MN3 3 0.456247 0.317966 0.254841 11.00000 0.02076
0.01088 =
0.03954 -0.00161 0.00608 -0.00006
MN4 3 0.064046 0.318290 0.258885 11.00000 0.01770
0.01177 =
0.04013 -0.00096 0.00270 -0.00291
MN5 3 0.103712 0.310492 0.457106 11.00000 0.02187
0.02206 =
0.03839 -0.00390 0.00212 -0.00034
MN6 3 0.381220 0.421973 0.549805 11.00000 0.02629
0.01616 =
0.03729 0.00000 0.00087 -0.00399
AS1 1 0.216884 0.589513 0.646409 11.00000 0.02081
0.01284 =
0.03526 0.00042 0.00483 -0.00059
AS2 1 0.233284 0.237383 0.641978 11.00000 0.02114
0.01622 =
0.03634 0.00155 0.00458 -0.00049
AS3 1 0.285640 0.486552 0.360846 11.00000 0.01828
0.01149 =
0.03503 -0.00108 0.00328 -0.00137
AS4 1 0.271660 0.146937 0.351330 11.00000 0.02078
0.01583 =
0.03724 -0.00069 0.00521 0.00072
O1 4 0.665795 0.324572 0.318465 11.00000 0.02580
0.01573 =
0.05030 -0.00198 -0.00757 -0.00599
O2 4 0.271843 0.558564 0.565914 11.00000 0.01973
0.02096 =
0.05632 -0.00090 0.01361 0.01192
O3 4 0.770856 0.514191 0.303457 11.00000 0.01903
0.00811 =
0.05115 0.00683 0.00283 0.00517

```

O4	4	0.964393	0.366292	0.358474	11.00000	0.02034
0.01587 =						
		0.03283	-0.00694	-0.00009	0.00202	
O5	4	0.228143	0.115860	0.628754	11.00000	0.02431
0.02774 =						
		0.03603	-0.00585	0.00648	0.00558	
O6	4	0.576519	0.227958	0.181001	11.00000	0.01965
0.01379 =						
		0.04517	-0.00568	0.01158	-0.00183	
O7	4	0.240909	0.302580	0.565303	11.00000	0.03283
0.02636 =						
		0.02695	-0.00905	0.00144	0.00093	
O8	4	0.891248	0.233534	0.196190	11.00000	0.00887
0.01812 =						
		0.04763	-0.00950	0.00688	-0.00450	
O9	4	0.159889	0.572801	0.337238	11.00000	0.02978
0.01110 =						
		0.04309	-0.00362	-0.00425	-0.00292	
O10	4	0.272737	0.394261	0.300834	11.00000	0.02936
0.02870 =						
		0.03318	0.00078	0.00412	-0.00546	
O11	4	0.260256	0.438932	0.442192	11.00000	0.02482
0.03863 =						
		0.02018	0.00010	0.00992	-0.00603	
O12	4	0.462212	0.539414	0.361563	11.00000	0.03125
0.01734 =						
		0.03083	0.00087	-0.00021	-0.00473	
O13	4	0.277203	0.217300	0.424301	11.00000	0.02238
0.02658 =						
		0.04539	-0.00488	0.00756	-0.00523	
O14	4	0.270962	0.026038	0.369552	11.00000	0.03305
0.01660 =						
		0.03136	0.00301	0.00268	0.00120	
O15	4	0.422536	0.176172	0.305649	11.00000	0.01405
0.01887 =						
		0.04015	-0.00354	0.00856	-0.00454	
O16	4	0.109817	0.173312	0.298300	11.00000	0.01998
0.02909 =						
		0.03431	-0.00310	-0.00975	-0.00248	
OW17	4	0.494252	0.456012	0.190527	11.00000	0.02299
0.01533 =						
		0.04186	-0.00227	0.00004	0.00365	
OW18	4	0.243240	0.274428	0.176898	11.00000	0.02455
0.01289 =						
		0.03985	-0.00955	0.00026	0.01126	
OW19	4	0.994599	0.453888	0.195376	11.00000	0.02373
0.00326 =						
		0.05311	0.00473	0.00596	-0.00485	
OW20	4	0.762277	0.137940	0.321162	11.00000	0.02560
0.01413 =						
		0.05813	-0.00647	0.01308	0.00570	
OW21	4	0.955365	0.386684	0.525763	11.00000	0.05563
0.10049 =						
		0.04180	-0.01372	0.00095	0.03840	

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OW22  4      0.949141      0.175103      0.464637      11.00000      0.04854
0.03747 =
          0.08906      0.00565      0.01316      -0.00064
OW23  4      0.523298      0.319174      0.489412      11.00000      0.02460
0.02403 =
          0.06469     -0.00265      0.00981      -0.00009
OW24  4      0.183631      0.338886      0.042223      11.00000      0.08552
0.04596 =
          0.06086      0.01187      0.00024      -0.00709
OW25  4      0.127460      0.026319      0.506281      10.50000      0.06740
0.03818 =
          0.02272      0.00585      0.00547      -0.00330
OW26  4      0.379296      0.028225      0.521695      10.50000      0.09170
0.04885 =
          0.05975     -0.00828      0.01851      -0.02014

```

HKLF 4

REM CL179

REM R1 = 0.1182 for 2513 Fo > 4sig(Fo) and 0.1365 for all 3407 data

REM 325 parameters refined using 0 restraints

END

WGHT 0.2000 0.0000

REM Highest difference peak 4.868, deepest hole -3.675, 1-sigma level 0.502

```

Q1    1    0.2164    0.1894    0.1478    11.00000    0.05    3.63
Q2    1    0.2581    0.3436    0.3584    11.00000    0.05    3.30
Q3    1    0.2183    0.3622    0.6386    11.00000    0.05    3.15
Q4    1    0.8818    0.4669    0.5513    11.00000    0.05    3.06
Q5    1    0.4053    0.1993    0.5414    11.00000    0.05    2.60
Q6    1   -0.0730    0.3064    0.2395    11.00000    0.05    2.28
Q7    1    0.2248    0.0275    0.6494    11.00000    0.05    2.21
Q8    1    1.0643    0.5131    0.2601    11.00000    0.05    2.14

```

;

_shelx_res_checksum 22871

_shelx_hkl_file

;

```

  0  -2   0   95.75   10.18
  0   2   0   99.58   10.13
  0  -2   0   67.60    6.68
  0   2   0   79.18    6.77
  1   1   1  2378.14   74.18
  1   1   1  2184.70   77.60
  1  -1   1  2243.12   92.89
 -1   1  -1  2124.60   70.00
  1  -1   1  2139.35   69.80
 -1   1  -1  2321.33   94.74
 -1  -1  -1  2209.02   64.39
 -1  -1  -1  2174.71   74.93
 -1   0   2   14.60    5.58

```

1	0	-2	20.43	5.31
0	-2	1	0.46	3.28
0	2	1	0.31	2.19
0	-2	-1	-0.81	3.15
0	2	1	4.38	4.58
0	-2	-1	0.12	4.54
0	2	-1	0.81	4.57
0	2	-1	1.35	3.32
0	0	-3	0.91	7.99
0	0	3	6.40	7.81
-1	0	-2	38.13	4.62
1	0	2	44.47	8.07
-1	-1	2	113.25	8.59
-1	1	2	76.89	8.79
1	-1	-2	83.10	8.88
1	1	-2	111.59	9.91
1	1	-2	100.90	11.15
1	-1	-2	87.02	7.71
-1	1	2	118.70	12.84
-1	-1	2	117.08	9.57
0	1	-3	490.10	21.61
0	1	3	483.23	33.29
0	-1	3	507.93	34.18
0	1	3	500.13	31.84
0	-1	-3	440.07	31.20
0	-1	3	470.92	33.91
1	-1	2	700.36	32.61
-1	1	-2	727.68	39.77
-1	-1	-2	661.06	33.30
-1	-1	-2	659.77	29.40
1	1	2	758.56	35.86
0	-2	-2	37.80	7.61
0	2	-2	54.53	8.21
0	2	-2	35.60	6.06
0	-2	-2	49.17	10.35
0	2	2	44.31	10.70
0	2	2	44.61	8.32
0	-2	2	39.44	8.21
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-1	2	0	100.37	10.13
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-1	2	0	86.15	8.01
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1	2	-1	996.47	29.88
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1	-2	1	262.75	18.02
-1	-2	-1	274.44	15.67

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-1	-2	-1	257.21	19.35
-1	2	-1	204.15	13.81
1	-2	1	257.85	21.92
1	2	1	269.33	19.89
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-1	1	3	68.18	7.84
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1	-2	2	68.28	13.07
1	-2	2	68.11	10.59
1	2	2	67.06	11.09
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0	3	0	4.18	4.15
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0	-1	4	5.17	8.24

0	1	4	-4.82	7.27
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2	-1	-3	188.78	20.44
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0	4	-2	289.03	32.22
0	4	-2	314.14	24.63
0	-4	2	277.33	23.63
0	-4	2	242.06	19.53
0	-4	-2	300.27	33.02
0	-4	-2	300.57	22.94
0	-4	-2	304.96	23.31
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-1	4	0	39.13	12.56
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-1	-4	0	44.15	6.05
-1	-4	0	54.36	13.68
1	4	0	36.29	7.92
-1	4	0	56.14	12.22
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2	3	0	4266.62	205.46
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-2	3	0	4291.99	195.19
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4	1	-1	227.64	9.23
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-2	-1	-7	318.26	42.59
-2	1	-7	272.94	44.00
2	-1	7	329.44	43.09
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4	1	0	98.96	14.60
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4	0	1	5.89	6.74
4	0	1	0.89	2.80
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-1	-5	5	-4.35	7.85
1	5	-5	14.67	10.75
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1	5	-5	-5.25	7.34
1	-5	-5	6.93	6.29
4	-1	-2	-1.25	5.12
4	1	-2	0.50	6.06
4	1	-2	8.33	8.68
4	1	-2	1.29	2.41
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-2	-3	-6	198.10	31.93
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2	-3	6	240.46	33.42
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1	6	-2	37.14	15.83
1	-6	-2	35.31	14.20
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2	-5	3	11.11	8.34
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-2	-5	-3	0.56	5.71
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4	1	1	264.98	14.12
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3	-4	-3	27.17	12.14
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3	4	-3	27.49	11.18
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2	4	5	1025.81	83.38
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3	-4	-4	7.11	7.44
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4	1	5	8.24	11.81
4	1	5	14.27	9.47
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3	7	5	415.80	39.45
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5	-5	-3	223.12	30.40
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-3	-6	8	-0.23	14.92
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-1	-8	-7	29.87	17.78
-1	-8	-7	44.58	28.03
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5	4	5	519.53	50.66
5	4	5	544.64	62.12
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5	0	-9	-2.59	12.20
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2	2	12	49.46	19.53
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2	2	12	53.12	18.40
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-2	5	11	7.94	18.47
2	-5	-11	10.26	18.25
6	0	3	8.12	4.69
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-2	-9	3	204.48	18.15
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3	5	9	107.68	32.24
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3	-5	9	201.53	26.06
3	-5	9	162.09	47.11
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6	3	-1	271.78	17.42
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4	-5	7	3.03	12.33
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-4	-5	-7	-8.27	13.57
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6	1	-5	384.43	36.28
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4	-1	-11	58.19	18.02
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2	1	-13	151.67	29.02
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4	1	-16	-34.24	43.56
4	-1	-16	52.86	24.59
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4	8	12	441.34	83.83
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7	7	6	-1.18	5.60
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7	-5	-15	236.51	66.33
-7	-5	15	114.64	44.57
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7	-5	-15	155.54	71.64
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-5	3	19	167.38	81.86
-5	-3	19	101.76	96.51
5	3	-19	92.81	83.72
-5	3	19	271.31	63.55
-5	-3	19	71.20	77.92
5	-3	-19	166.90	100.46
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7	-8	-12	520.55	156.37
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4	-11	-15	-26.59	37.03
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8	1	14	21.02	34.47
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